Dimensional Change of Dental Apatite-based Glass Ceramic : Effect of Different Investment Types

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ABSTRACT

The dimensional change of castable inlay of dental apatite-based glass ceramic, that is, 20 wt% CaO, 10 wt% P_2O_5, 10 wt% MgO, 10 wt% Al_2O_3 and 50 wt% SiO_2 was examined by marginal gap when cast into three types of investments, such as phosphate-bonded, ethyl silicate-bonded cristobalite-quartz and magnesia-alumina-based investments. Castable glass ceramic inlay margin in the former two was obtained with smaller gap under higher value of porosity within the mould while cast inlay in the latter one was not completed by vacuum-pressurized casting machine. In order to use phosphate-bonded investment as a mould with higher porosity, colloidal silica suspension was diluted with distilled water. It was confirmed that the magnitude of marginal gap in apatite-based glass inlay decreased effectively using phosphate-bonded investment mixed by aqueous colloidal silica.

INTRODUCTION

Dental castable glass ceramics have been developed as restorative materials because of their ease in casting and esthetic merit, and their wax patterns made by lost wax technique were invested in the investment mould[1,2]. Glass ceramic crown does require that appropriate expansion value of investment compensates the shrinkage of castable glass ceramic when cast into investment mould[1-3]. The ethyl silicate-bonded investment (included alpha cristobalite and quartz powders), developed for castable glass materials, was suitable to compensate castable ceramic crown of developed apatite-based glass ceramic[4]. Setting and thermal expansion values (total expansion) of dentaal investments were controlled by adjusting a pH value of mixing solution[5,6]. The magnitude of total expansion was also improved by investment powders (alpha cristobalite and/or quartz) incorporated in investment mould, or included phosphate compound[7-10]. As an attempt to cast dental glass ceramic inlay, there have been only a few reports[3,11], in which the investments tested were not available to cast glass ceramic inlay. It is thus important to examine the property of dental cating investment to cast glass ceramic inlay. This study is to examine the marginal gap of castable apatite-based glass ceramic when cast into different type of investment moulds.

MATERIALS AND MEHODS

1. Materials

Dental apatite-based glass ceramic was an experimental one, containing 20 wt% CaO, 10 wt% P_2O_5, 10 wt% MgO, 10 wt% Al_2O_3 and 50 wt% SiO_2. B_2O_3 and CaF_2 were also added by a very small amount (0.01 wt%) to the mother glass[12,13]. The ceramic was cast into the investment moulds using vacuum-pressurized casting machine (PROTOTYPE II, J. Morita Co, Kyoto)[14], as illustrated in Figure 1. Four dental investments, such as two phosphate-bonded investments (I1; G C Co, Kyoto, I3; Olympus Co, Tokyo) and newly developed ethyl silicate-bonded cristobalite/quartz investment (I2)[4], and magnesia/alumina-based investment (M1; J. Morita Co), examined in this study were listed in Table 1. Investment powders in I1, I2 and I3 were alpha cristobalite and quartz, and M1 contained magnesia and alumina (mixed with a special liquid without colloidal silica). For I1 and I3, the powders were mixed with colloidal silica. I2 investment powder was mixed with mixture (A and B)[4] (A; pH-adjusted silica sol, 16 mL and B; aqueous ammonia, 1 mL). A colloidal silica-to-water ratio used in this study was 10/0, 7/3, 5/5, 3/7 and 0/10. The liquid-to-powder ratio was 0.32 (I1, I3), 0.34 (I2) and 0.16 (M1), respec-

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tively, when the investment powders were mixed with the liquid.

2. Methods

(1) Setting and thermal expansion values

The setting behaviour was determined as the linear dimensional change that occurs as the investment sets in the stainless steel (25 mm in diameter and 5 mm long) through lined with Kaolin material (Dentsply Co, York, USA), with travelling microscope (Seiki-shya, Tokyo).

The thermal expansion behaviour of the samples, 5 mm in diameter and 12 mm long, was examined with a thermal analyzer (TMA Thermoflex, Rigaku Co, Tokyo), using the following condition: heating rate, 10° C/min and heating ranges, 25 to 800° C.

(2) Density and particle size measurements

The value of true density (D_t) was measured by a pycnometric method, employing a Hubbard-type specific gravity bottle (Shibata Co, Tokyo). Theoretical porosity was thus calculated as follows.

$$\text{Porosity (\%)} = (1 - D_b/D_t) \times 100$$

, where D_b means bulk density (gram/cm^3) and Dt true density gram/cm^3).

The distribution of particle sizes of the investments were shown in Fig. 2 (Particle size analyzer SALD-2000, Shimadzu Co, Kyoto). The powders were immersed in the solvents, centrifuged and counted according to sedimentation method.

(3) Water absorption as uptake

The amount of water absorption under saturation when immersed in distilled water was measured. Before measuring it, the samples were placed in air after immersion and continuously placed until the constant weight was obtained. The magnitude of water absorption was calculated in the following equation.

$$\text{Water absorption (\%)} = ((W_3 - W_1)/W_1) \times 100$$

, where W_1 and W_3 mean dry weight (mg) when kept at room temperature before testing and saturated weight after immersion in water (mg), respectively.

(4) Marginal gap measurement

Simple type inlay of castable glass ceramic (8.0 and 7.0 mm; diameter at upper and lower surfaces and 6.1 mm height) was formed with wax (Blue inlay wax, GC Co, Tokyo) at 60° C, and wax pattern was compressed (1.4 Mpa) and held for 10 min within the stainless steel die. The pattern was stored at room temperature for at least 2 hours to allow release of residual strain within the wax. A 4.0 mm diameter × 10 mm long sprue was luted to the centre of upper surface and the wax pattern was removed from the steel die mould, mounted on the sprue former and was invested immediately within a steel ring (32 mm diameter × 50 mm long; Fig. 1(a)). Casting operation was carried out as written in schematic figure (Figs 1(b) to (d)).

After casting the investment powders were removed using ultrasonic cleanser, and the value of marginal gap was directly measured as a clearance value between castable inlay and steel die (samples = 10 pieces for each investment mould). Statistical analysis of marginal gap was carried out using Student *t'* test.

RESULTS

The amount of total expansion (setting and thermal expansion) was 2.4% for I1, I2 and I3 investments, whereas M1 had 1.5%. The density values measured were 2.46, 2.34, 2.24 for I1, I2, I3, and 3.18% for M1, respectively

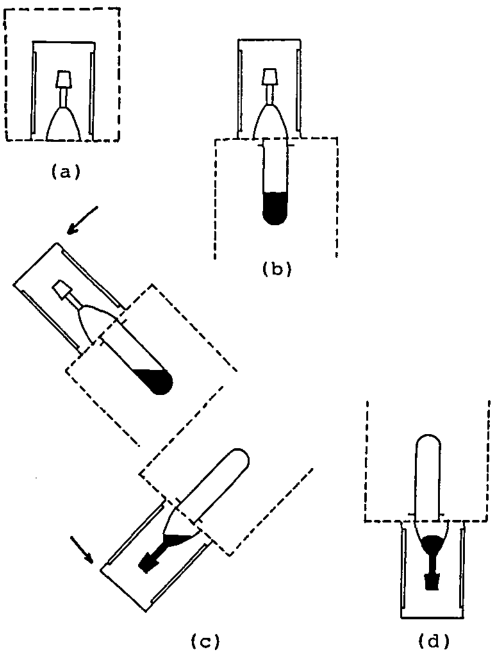

Figure 1 Schematic figure for casting procediures (vacuum-pressurized casting machine). (a) Investment mould in furnace heated to each condition (see text for the detail), (b) ring set, (c) rotation of the mould and (d) casting into the mould.

Table 1 Investment types tested; Composition and true density measured.

Code	Investment	Refractory Powder	Density*
I1	Ceravest Quick	Cristobalite and quartz	2.46 (0.014)
I2	Bioram Ethylsilicate		2.34 (0.019)
I3	Maizoudon		2.24 (0.086)
M1	Titavest CB	MgO and Al_2O_3	3.18 (0.050)

*True density measured

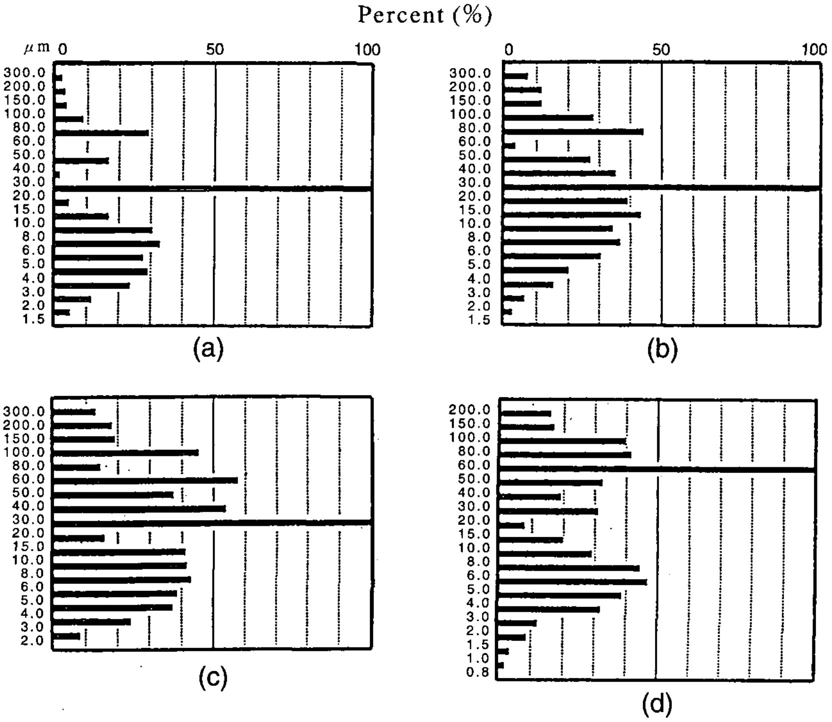

Figure 2 Distribution of particle size in the investment powders tested. (a) I1, (b) I2, (c) I3 and (d) M1. See text for key.

(Table 1).

Fig. 2 shows particle size distribution of investment powders tested. For I1, alpha quartz had 4 to 10 µm and alpha cristobalite 20 to 30 µm as the median value. For I2, alpha quartz had 5 to 15 µm and alpha cristobalite 20 to 30 µm. For I3, alpha quartz and cristobalite had 4 to 15 µm and 20 to 30 µm, respectively. For M1, alimina and magnesia had 3 to 8 and 50 to 60 µm, respectively. The results show that alpha cristobalite size is similar among I1, I2 and I3 while magnesia particle size in M1 is greater than those median sizes (20 to 30 µmi) in I1, I2 and I3.

Figs. 3 and 4 show theoretical porosity and water absorption of the investment tested. The porosity increased with increasing bulk density. More increased porosity in I1 than I2 and I3 was found while the porosity in M1 with greater predicted bulk density than those of I1, I2 and I3 was supposed to be lower than in I1, I2 and I3. Water absorption in I1, I2 and I3 (21 to 25%) was greater than in M1 investment (14%), and also those values in I1 (7/3, 5/5, 3/7 and 0/10; colloidal silica/water) were similar to, or greater (28%) than, I1 (10/0). Thus, I1 investment which showed greater porosity than in I2 and I3 was applied additionally for diluted silica mixture.

Figs. 5(a) and (b), respectively, show the value of marginal gap when cast into the investment moulds tested. Smaller value of marginal gap in I1 than I2 annd I3 was obtained. The use of diluted collodal silica to mixing

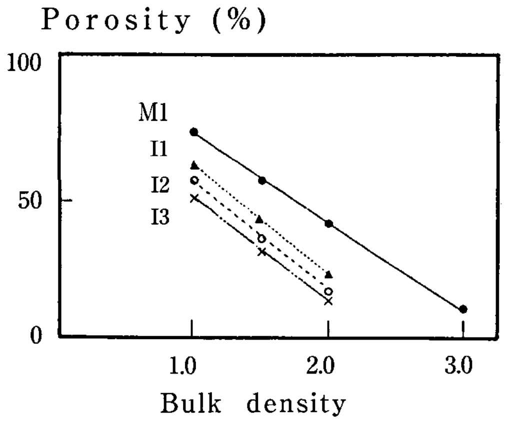

Figure 3 Theoretical porosity with respect to bulk density of the investments tested. See text for key.

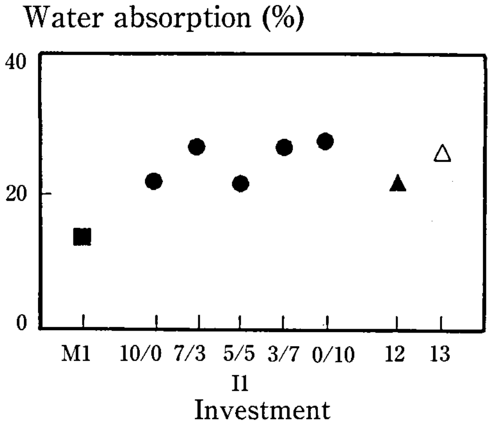

Figure 4 Water absorption in the investment tested (I1, I2, I3 and M1). I1 investment was mixed by colloidal silica diluted with water.

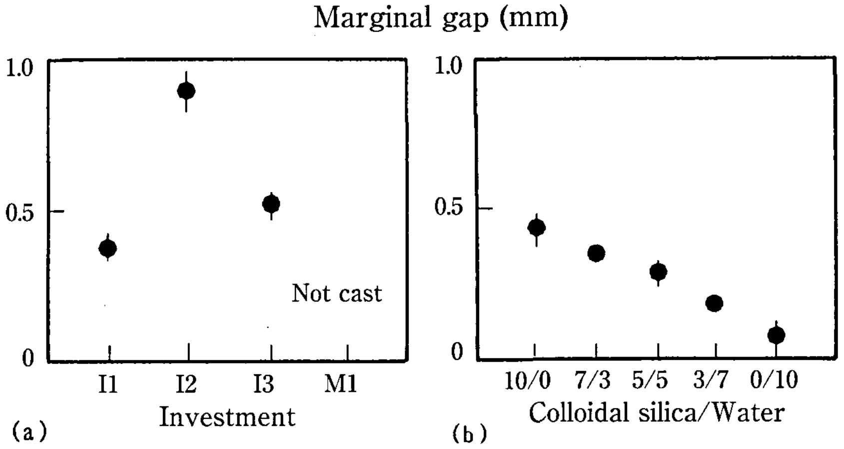

Figure 5 Marginal gap of castable glass inlay. (a) I1, I2, I3 and M1 investments, (b) I1 investment mixed with diluted colloidal silica.

solution exhibited the decreased marginal gap, and marginal gap of cast inlay when mixed by 7/3 solution (colloidal silica/water) was smaller significantly than those 10/0, 7/3 and 5/5 ($p < 0.01$).

DISCUSSION

Using 20 wt% CaO /10 wt% P_2O_5/10 wt% MgO/10 wt% Al_2O_3/50 wt% SiO_2 ceramic, dental castable crown was cast into ethyl silicate-bonded investment mould, which was noted by I2 (ethyl silicate-bonded) in this study[3]. After ammonium carbonate aqueous solution was added to each silica sol, I2 mould hardened associated with the start of sol-gel reaction (pH = 8.0 to 8.5)[3,4]. In order to meet the purpose to obtain castable glass ceramic inlay, the investment moulds for casting of apatite-based glass ceramic should be needed. To apply glas ceramic to castable glass inlay, commercial investments were used. This is because that their investment types (I1, I2, I3) tested have the same expansion value (total; 2.4%) and an ability to cast the glass ceramic is needed for the investment moulds[1,15]. The effect of the castability was dependent upon porosity in the mould related to filler size, and the increased amount of porosity exhibited more increased water absorption amount within the mould (Figs. 3, 4).

Because apatite-based glass ceramic was 1160° C as the melting temperature[3,13,16], it is observable that adhering oxides form little on the cast surface and the ethyl silicate-bonded mould gave complete cast inlays. Homes et al[15] reported that ceramic cast crown (mica-based) was used with phosphate-bonded investment and a fit comparable to that of metal crowns was also resulted. The results showed that surface smoothness and marginal fit were related clearly. Thus, this study examined the diffrence of marginal gap in castable glass inlay when used by three types of dental investments and mixed by diffrent colloidal silica-to-water ratio for I1 (Table 1 and Fig. 5).

In the case of cast crown in the galss state, a volumetric contraction during cooling after the cast affected marginal gaps[17,18]. Thus, marginal gap of cast inlay was determined by shrinkage of volumetric contraction as follows, according to the concept described by the same authors[19],

Total expansion = (setting and thermal expansion) = 180 μm
Total shrinkage = (delta T) (alpha) (l_0) = 70 μm
Difference = 110 μm

, where delta T = solidification temperature — room temperature

= 1500–25 = 1475° C[3,13]

alpha = 63×10^{-7}/° C (thermal expansion coefficient)[13]

l_0 = 7.5 mm ((8.0 + 7.0)/2) as mean diameter

The difference between investment expansion and thermal shrinkage of the castings was 110 μm for castable glass inlay. The predicted value was close to the minimum value measured (Fig. 5 (b)). The much smaller of thermal shrinkage of the castings, or decreased total expansion, or appropriate combination of their values.

In casting the present glass ceramic into ethyl silicate-bonded investment mould, the interface between mould and glass ceramic was affected by particle sizes of refractory fillers in the form of alpha cristobalite and alpha quartz[15]. The distribution of fillers (alpha quartz) was completely different among the invetsments tested (I1, I2, I3), and M1 investment contained greater size of magnesia and smaller size of alumina. It is supposed that their particle distribution might affect the magnitude of water absorption and theoretical porosity (Figs 2 and 4). The investment type and, especially, concentration of colloidal silica affected the value of marginal gap of apatite-based glass ceramic, aand the smallest margin (0.1 mm) also agreed with the value which was evaluated in this section.

CONCLUSION

The present study examined the possibility for castable glass inlay, showing that selected investment powders and collidal silica dilution were effective to give smaller marginal gap between steel die and its inlay. This result supported effectively to improve the margin gap of castable glass inlay.

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REFERENCES

1) Adair, P.J. and Grossman, D.G.: The castable ceramics crown. *Int. J. Perio. Resror. Dent.* **2**, 33–45, 1984.
2) Matsui, A.: The developmental study of glass-ceramics on bioramics field. *The Quintessence* **7**, 105–120, 1988.
3) Nomura, Y., Taira, M., Wakasa, K. and Yamaki, M.: Effect of the addition of magnesia powder into the exclusive investment on the adaptability of calcium-phosphate-bonded castable glass-ceramic inlays. *Hiroshima Daigaku Shigaku Zasshi* **22**, 363–368, 1990.
4) Wakasa, K. and Yamaki, M.: Application of silica-sol investment to dental castable ceramics. *J. Mater. Sci. Letters* **12**, 1897–1899, 1993.
5) Finger, W. and Jorgensen, K.D.: An improved dental casting investment. *Scand. J. Dent. Res.* **88**, 278–284, 1980.
6) Finger, W. and Kota, K.: A modified phosphate-bonded casting investment. *Scand. J. Dent. Res.* **90**, 243–248, 1982.
7) Wakasa, K. and Yamaki, M.: A modified dental cast investments for nickel-base alloy: a preliminary study. *J. Mater. Sci. Letters* **10**, 1093–1094, 1991.
8) Mori, M., Yanagihara, T., Asai, T., Itoh, S. and Mukai, M.: Studies on investment with carbon Part 2. *Aichi-gakuin Daigaku Shigaku Zasshi* **15**, 298–305, 1977.
9) Mori, T.: The α- and β- forms of calcium sulphate hemihydrate. *J. Dent. Res.* **65**, Abst. No. 38, 1986.
10) Mori, T.: Thermal behavior of the gypsum binder in dental casting investments. *J. Dent. Res.* **65**, 877–

884, 1986.

11) Honda, K.: Study on investments for castable ceramics Part 1. Thermal expansion of investments. *Nihon Shika Hozongaku Zasshi* **37**, 50–55, 1994.

12) Wakasa, K., Matsui, H. and Yamaki, M.: A thermoanalytical study on non-asbestos ring-lining materials for dental application. *J. Mater. Sci. Mater. Med.* **1**, 207–210, 1990.

13) Wakasa, K.,Yamaki, M. and Matsui, A.: Thermal treatment and crystalline structure in dental cast ceramic material. *J. Mater. Sci. Mater. Med.* **3**, 235–238, 1992.

14) Wakasa, K. and Yamaki, M.: Thermal behaviour of dental investment during setting. *J. Oral Rehabil.* **22**, 371–375, 1995.

15) Holmes, J.R., Sulik, W.D., Holland, G.A. and Bayne, S.C.: Marginal fit of castable ceramic crowns. *J. Prosthet. Dent.* **67** 594–599,1992.

16) Wakasa, K. and Yamaki, M.: Mould interface observation of castable ceramic crowns in casting and ceramming. *Hiroshima Daigaku Shigaku Zasshi* **25**, 494–500, 1993.

17) Dicor research report: *Fit of Dicor castable ceramic crown*, Dentsply Int, York, Pa, USA, 1986.

18) Ishida, H., Nahara, Y. and Hamada, T.: Dimensional accuracy of castable apatite ceramic crowns: The influence of heat treatment on dimensional changes and distortion of crowns. *J. Prosthet. Dent.* **68**, 270–283, 1992.

19) Wakasa, K., Ogino,S., Yoshida,Y. and Yamaki, M.: Accuracy of apatite-based glass ceramic in dental cast ethyl silicate-bonded investment: Theoretical consideration. *Hiroshima Daigaku Shigaku Zasshi* **28**, 5–10, 1996.